

BOND SENSITIVITY TO SILICONE CONTAMINATION

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Introduction

Currently during fabrication of the Space Shuttle booster rocket motors, the use of silicone and silicone-containing products is prohibited in most applications. Many shop aids and other materials containing silicone have the potential, if they make contact with a bond surface, to transfer some of the silicone to the substrates being bonded. Such transfer could result in a reduction of the bond strength or even failure of the subsequent bonds. This concern is driving the need to understand the effect of silicones and the concentration needed to affect a given bond-line strength. Additionally, as silicone detection methods used for materials acceptance improve what may have gone unnoticed earlier is now being detected. Thus, realistic silicone limits for process materials (below which bond performance is satisfactory) are needed rather than having an absolute "no silicone permitted" policy.

Experimental

Testing was conducted in which D6ac steel specimens were initially cleaned using an aqueous spray-in-air process with a BrulinTM 1990 solution, which was followed by grit blasting the metal surfaces before contamination application. The silicone, DC200 (a polymethylsiloxane--1,000 centistokes viscosity) manufactured by Dow Corning was dissolved in methylchloroform to the appropriate level to obtain the desired concentration on the bond surface after spray application. The silicone was sprayed on the hardware to the approximate desired level using a Sono-Tek ultrasonic spray system. More than one pass in the Sono-Tek was required for the higher concentrations. The amount of silicone applied to the hardware was determined both gravimetrically and with the use of a solvent flush and FTIR comparison with a previously produced master curve. The flush technique was used for results less than 5 mg/ft² and the gravimetric technique was used for results in excess of 5 mg/ft².

After applying the silicone to the hardware, the specimens were allowed to stand for three to five days to allow the silicone to more fully disperse over the bond surface. Control specimens, which were sprayed with me-

thylchloroform only, and staged with the contaminated samples were also prepared.

The evaluation of the silicone contamination effects on bondlines included two epoxy resin adhesives EA 913NA and TIGA 321, a Rust-Oleum white topcoat paint with a zinc rich primer, and an asbestos-silica filled NBR rubber bonded to steel by vulcanizing using Chemlok 205/233. The adhesives are used on the Space Shuttle rocket motor booster nozzle; the paint/primer on the exterior of the rocket motor cases and the Chemlok is used to vulcanize internal insulation to the rocket motor cases.

EA 913NA and TIGA 321

These studies used D6ac steel tensile adhesion buttons bonded to a steel panel and tapered double cantilever beam (TDCB) specimens. During normal usage, the adhesives are used with a silane primer, Silquest A-187 based; however, to assess the degree to which the silane primer helps overcome adverse situations, only half of the samples were primed. All of the buttons were grit blasted just prior to bonding and were primed to force the failure to the button-panel interface. The button-to-panel bondline used 0.030-inch thick Delrin spacers and the TDCB's used 0.050-inch thick Teflon spacers to control the bondline thickness. After assembly of the specimens, they were placed in fixtures that allowed pressure to be applied across the specimen bond surfaces. The adhesives were cured at 105 ± 5°F with EA 913NA adhesive being cured for 42 to 45 hours and the TIGA 321 adhesive for 48 hours minimum. The button-to-panel specimens were tested at 0.05 inches/minute and TDCB's at 0.005 inches/minute at 72 ± 2°F. Twelve primed and twelve unprimed spaces on the panel for the buttons were used for each data point and four primed and four unprimed TDCB sets were used for each of the fracture toughness points.

Rust-Oleum

These studies used D6ac steel panels, which were sprayed with the Rust-Oleum primer, cured for 24 hours minimum, sprayed with the topcoat and cured for an additional 24 hours minimum. The paint was lightly abraded, cleaned with a methylchloroform dampened cloth, and tensile adhesion buttons, which were grit blasted and silane primed just before use, were bonded to the panel using adhesive EA 934NA. During normal usage, no silane

primer is used with this bond; however, to assess the degree to which the primer helps overcome adverse situations, one panel at the highest silicone contamination level had silane primer applied to it prior to priming and painting. All of the buttons were grit blasted just prior to bonding and were silane primed to force failure to the paint-panel interface. The button-to-panel bondline used 0.030-inch thick Delrin spacers. After assembly of the specimens, the adhesive was cured five days minimum at ambient room temperature. The button-to-panel specimens were tested at 0.05 inches/minute at $72 \pm 2^\circ\text{F}$. Twelve buttons were used for each data point.

NBR/Chemlok 205/233

These studies used D6ac steel panels and beveled tensile adhesion buttons (the buttons were grit blast just prior to use), which were sprayed with the Chemlok 205 primer, dried for 60 minutes minimum, sprayed with the adhesive Chemlok 233 and dried for an additional 30 minutes minimum. The specimens were then assembled in a mold that allows for seven 45-degree peel specimens and eight tensile adhesion button specimens. The specimens were vacuum bagged and the rubber vulcanized and bonded with a step-wise cure that cures the rubber for six hours at 290°F and a pressure of 100 psi for six hours. After cure, the 45-degree peels were cut into 1-inch wide specimens. The button-to-panel specimens were tested at 0.5 inches/minute and the 45-degree peels at 2.0 inches/minute at $72 \pm 2^\circ\text{F}$.

Results and Discussion

The results of testing of the EA 913NA adhesive for both the tensile adhesion button and TDCB testing are shown in Figure 1. The results show that the tensile adhesion results are less affected by the silicone than are the fracture toughness results. Additionally the silane primer has larger comparative effect on improving the bond strength of the tensile adhesion testing than on the fracture toughness results. Previous tensile adhesion testing with this adhesive has shown that most of the experimental variation is caused by lot-to-lot variation rather than other variables in the fabrication process and that the silane primer generally enhances the bond strength when process problems are encountered [1].

The results of testing of the TIGA 321 adhesive for both the tensile adhesion button and TDCB testing are shown in Figure 2. The results show that the tensile adhesion results are also less affected by the silicone than are the fracture toughness results. Additionally the silane primer has a slight effect on improving the bond strength of both the tensile adhesion and fracture toughness results.

The results of testing of the Rust-Oleum paint for tensile adhesion button testing are shown in Figure 3. The results show that the tensile adhesion results are affected by the silicone. The one test where silane primer was used

had a comparatively large effect on improving the bond strength.

The results of testing of the NBR—Chemlok 205/233-steel bond for both the tensile adhesion button and 45-degree peel testing are shown in Figure 4. The results show that for the tensile adhesion results the silicone contamination has little effect on the bond strength until a silicone contamination level of 10 mg/ft^2 are applied, whereas the 45-degree peel testing results show a tolerance for the silicone of up to 30 mg/ft^2 .

Conclusions

The results indicate that for the epoxies and paint, silicone levels as low as 0.1 mg/ft^2 affected the bond strength. The use of a silane primer helped to ameliorate the effects of the silicone contamination, but insufficiently to be able to ignore the effect on the bond strength. The vulcanized rubber bond tolerated silicone levels that were considerably higher. Testing shows that silicone controls are necessary for critical bonds and should be considered a potential cause when investigating unexpectedly low bond performance.

References

1. G. A. Caldwell, "Bond Lab Certification Program" 44th International SAMPE Symposium, 1999, Book 1, pp. 562-571

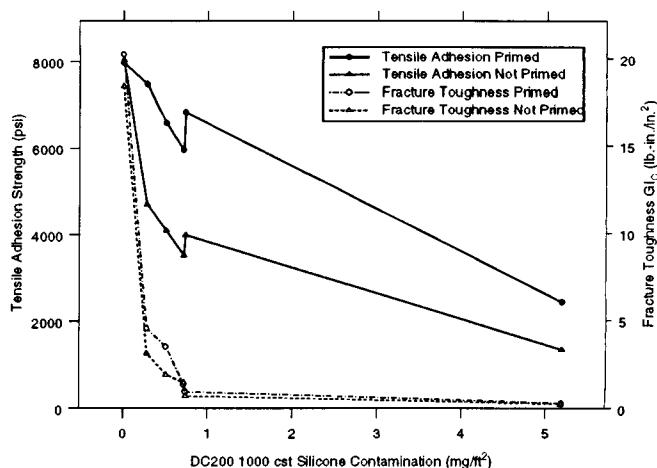


Figure 1. Tensile Adhesion Strength and Fracture Toughness of EA 913NA-to-D6ac Steel Bond Surface Contaminated with DC200 (1000 cst.)

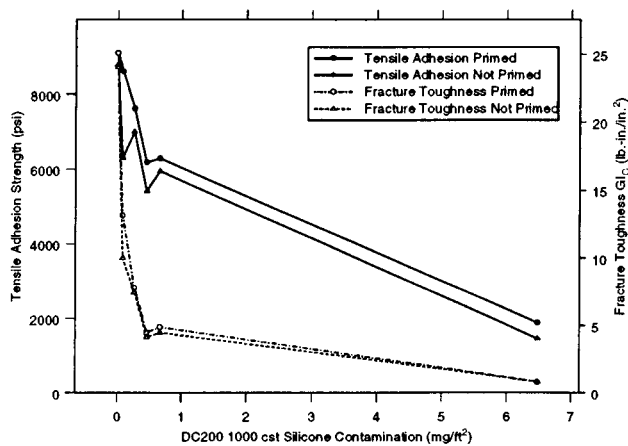


Figure 2. Tensile Adhesion Strength and Fracture Toughness of TIGA 321-to-D6ac Steel Bond Surface Contaminated with DC200 (1000 cst.)

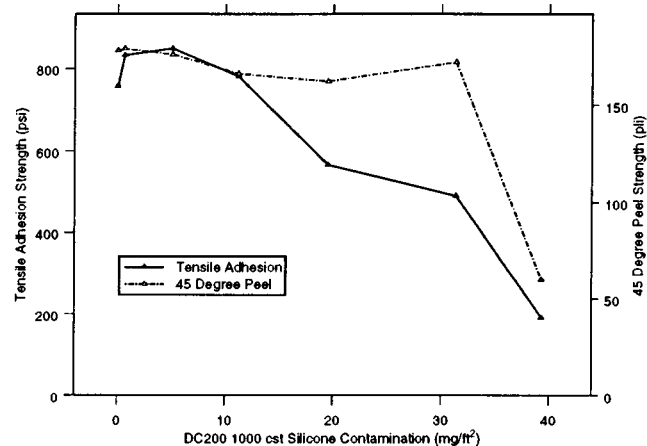


Figure 4. Tensile Adhesion and 45-Degree Peel Strength of NBR—Chemlok 205/233-to-D6ac Steel Bond Surface Contaminated with DC200 (1000 cst.)

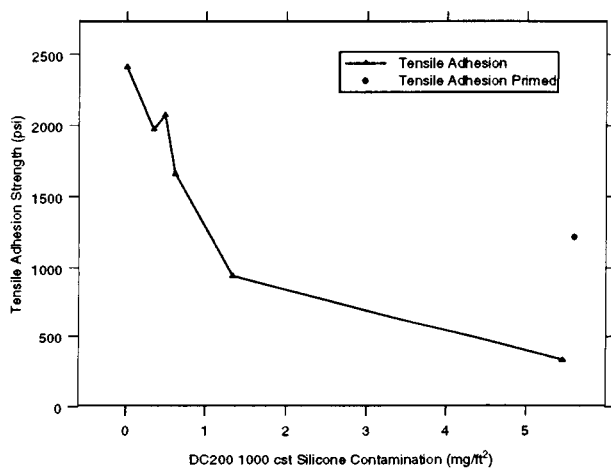


Figure 3. Tensile Adhesion Strength of Rust-Oleum Paint-to-D6ac Steel Bond Surface Contaminated with DC200 (1000 cst.)